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BUREAU OF STANDARDS
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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR
VARNISH, SHELLAC

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 376

This specification was officially promulgated by the Federal Specifications Board on January 25, 1926, for the use of the departments and independent establishments of the Government in the purchase of varnish, shellac.

[The latest date on which the technical requirements of this specification shall become mandatory for all departments and independent establishments of the Government is April 26, 1926. They may be put into effect, however, at any earlier date, after promulgation.]

CONTENTS

	Page
I. General specifications.....	1
II. Types.....	1
III. Material.....	1
IV. General requirements.....	2
V. Detail requirements.....	2
VI. Methods of sampling and testing.....	2
VII. Packing and marking.....	6
VIII. Notes.....	6

I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. TYPES

Shellac varnish shall be furnished as "Orange," type 1 or 2, and as "Bleached," type 1 or 2. Each type shall be furnished as light, medium, or heavy body, as specified in the contract.

III. MATERIAL

Shellac varnish shall be made by "cutting" the specified type of shellac, the manufactured product of stick lac (the secretion of the *Tacchardia Lacca*) in 95 per cent specially denatured alcohol, formula No. 1 of the United States Internal Revenue Bureau.

IV. GENERAL REQUIREMENTS

There are no general requirements applicable to this specification.

V. DETAIL REQUIREMENTS

The nonvolatile matter in shellac varnish shall conform to the requirements as given below for the respective types:

	Orange		Bleached	
	Type 1	Type 2	Type 1	Type 2
Iodine number (maximum).....	18.0	24.5	10.0	10.0
Matter insoluble in hot 95 per cent alcohol (maximum) per cent..	1.75	3.00	1.00	.10
Wax (maximum).....do.....	5.50	5.50	5.50	.20
Ash (maximum).....do.....	1.00	1.00	1.00	.30

Color, when specified, shall be no darker than a sample of shellac varnish mutually agreed upon by the buyer and seller.

The body of the respective types of varnish shall be based upon the percentage of nonvolatile matter determined as in section VI-2-(a):

	Orange, type 1 or 2	Bleached, type 1 or 2
Minimum allowable percentage of nonvolatile matter:		
Light body varnish.....	33.4	32.3
Medium body varnish.....	36.4	35.3
Heavy body varnish.....	39.1	37.9

VI. METHODS OF SAMPLING AND TESTING

Deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional information to ascertain whether the material meets the specification.

1. SAMPLING.—It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1,000 packages be taken as representative of the whole. Whenever possible an original unopened container shall be sent to the laboratory, and when for any reason this is not done the inspector shall thoroughly mix the contents of the container sampled, transfer not less than 1 quart to a clean dry glass bottle, which must be nearly filled with the sample, securely stoppered with a new clean cork or well-fitting cover or cap, sealed and distinctly labeled by the inspector. The inspector should take a duplicate from the container sampled, to be held for check in case of dispute, and when requested should take a sample for the seller.

2. LABORATORY EXAMINATION.—(a) *Nonvolatile matter*.—Place a portion of the thoroughly mixed sample in a stoppered bottle or weigh-

ing pipette. Weigh the container and sample. Transfer 1.25 g (± 0.25 g) of the sample to a weighed flat-bottomed metal dish about 8 cm in diameter (a friction-top can plug). Weigh the container again and by difference calculate the exact weight of the portion transferred to the weighed dish. Heat the dish and its contents in an oven maintained at 105 to 110° C. for three hours, cool, and weigh. From the weight of the residue left in the dish and the weight of the sample taken calculate the percentage of nonvolatile residue.¹

(b) *Iodine number of the nonvolatile residue.*—Transfer 1.5 cc of the shellac varnish by means of a pipette to a flat-bottomed glass or porcelain dish at least 8 cm in diameter. Add 1 to 2 cc of 95 per cent alcohol to spread the varnish evenly over the bottom of the dish. Heat the dish and contents at 85 to 90° C. for one-half hour. Scrape the residue from the dish and place 0.2 g in a 250 cc dry bottle of clear glass with a ground-glass stopper, add 20 cc of glacial acetic acid (see Reagents), and warm the mixture gently on top of a hot-water bath until solution is complete (except for the wax). A pure shellac is rather difficultly soluble; solution is quicker according to the proportion of rosin present. Add 10 cc of chloroform (see Reagents), and cool the solution to 21.5 to 22.5° C. The bottles should be allowed to stand half immersed in a shallow pan of water, well insulated or equipped with a suitable thermostat, at least 30 minutes at 21.5 to 22.5° C. before the Wijs solution (see Reagents) is added. Add 20 cc of Wijs solution (which shall be at a temperature of 21.5 to 22.5° C.) from a pipette, having a rather small delivery aperture (about 30 seconds). Close the bottle, place it back into the pan of water, and note the time. The bottles must be kept half immersed in water at 21.5 to 22.5° C. during the one hour that the shellac is exposed to the Wijs solution. Agitate the bottles occasionally during that hour.

NOTE.—If a number of samples are being run, at least five minutes should be allowed between the additions of the Wijs solution.

After exactly one hour add 10 cc of freshly prepared 10 per cent potassium iodide water solution (see Reagents), washing into the bottle any Wijs solution on the stopper with the same. Titrate the solution immediately with the 0.1 *N* sodium thiosulphate solution (see Reagents), allowing the solution to run in slowly (about 25 to 30 cc), with vigorous shaking until the solution becomes a straw color. Now add 15 cc of freshly prepared starch solution (see Reagents) and finish titrating. The end point is sharp, as the reaction products of shellac remain dissolved in the chloroform; any color returning after one-half minute or so is disregarded.

¹ The method described in Tentative Methods of Testing Shellac Varnish, published by the A. S. T. M. in 1925 may be followed, but the calculation shall be based on solids determined without any allowance for an assumed moisture content in the shellac, since this has been allowed for in the specification.

A blank determination shall be run at the same time on the reagents. The blank is necessary on account of the well-known effect of temperature changes on the volume and possibly loss of strength of the Wijs solution.

From the volumes of thiosulphate solution required for the sample and the blank, respectively, and the iodine value of the thiosulphate solution, calculate the iodine number (centigrams of iodine per gram of sample) of the shellac.

The analyst should run in parallel a sample of pure shellac of known iodine number as a check upon the reliability of the Wijs and thiosulphate solutions.

(c) *Material insoluble in hot 95 per cent alcohol.*²—Thoroughly shake the sample and at once place a portion of the well-mixed sample in a stoppered bottle or weighing pipette. Weigh the container and sample, thoroughly mix by shaking, and transfer approximately 5 cc of the sample to a small beaker. Weigh the container again and by difference calculate the exact weight of the portion transferred to the weighed dish. Heat the shellac varnish in the beaker with 25 cc of 95 per cent alcohol (see Reagents). Prepare a Gooch crucible with an abestos pad in the customary manner and dry to constant weight. Arrange the crucible for filtration by suction and pour sufficient boiling alcohol through it to thoroughly heat the crucible.

NOTE.—A cold crucible will congeal the wax and hinder filtration.

Immediately filter the boiling shellac solution, using suction, transfer the insoluble matter from the beaker to the crucible, using a "policeman" if necessary and a wash bottle containing hot alcohol. Wash the residue in the crucible with boiling alcohol five times, nearly filling the crucible each time.

NOTE.—It will be necessary to shut off the suction momentarily to fill the crucible.

Wash off any film of shellac on the sides or bottom of the crucible with hot alcohol and dry to constant weight in an oven at 105 to 110° C. The weight of the residue in the crucible multiplied by 100 and divided by the weight of the nonvolatile matter in the sample taken is the percentage of material insoluble in hot alcohol.

(d) *Wax.*—Thoroughly mix the sample and at once transfer a weighed portion of approximately 30 g to a tall cylindrical vessel. Dilute the varnish with 180 cc of 95 per cent alcohol (see Reagents) at a temperature of 24° C. $\pm$ 3° C. and thoroughly mix. Allow the solution to stand for several hours, preferably overnight in a tall cylindrical vessel maintained at a temperature of 24° C. $\pm$ 3° C. until the

² "The continuous extraction method" (A. S. T. M. Standards, 1924, p. 829, Method D29-24) may also be used.

wax has settled to a small layer at the bottom of the container. Decant the clear solution through a 12.5 cm folded filter paper, taking care not to disturb the wax layer. Finally, transfer the wax to the filter, using 25 cc of alcohol at the prescribed temperature. Wash the container and the wax with four 15 cc portions of alcohol. The final washings should be colorless. Allow most of the alcohol to evaporate from the filter paper and dissolve the wax by pouring successive small portions of boiling chloroform (see Reagents) through the paper. Also wash the container with boiling chloroform and pour on the filter. Collect the filtrate, which should contain all the wax, in a tared dish, evaporate the chloroform and dry to constant weight at 105 to 110° C. Weigh the wax residue and calculate the percentage of wax based on the weight of nonvolatile residue in the sample of shellac varnish taken.

The removal of the wax from the filter paper may also be accomplished in any suitable continuous extraction apparatus.

(e) *Ash*.—Transfer an accurately weighed portion of 5 to 6 g of the sample to a weighed porcelain crucible and evaporate most of the alcohol on the steam bath. Ignite at as low a temperature as possible until all organic matter has been destroyed. Cool, weigh, and calculate the percentage of ash based upon the weight of nonvolatile material in the sample of varnish taken.

(f) *Color*.—Compare the color of the sample under examination with the color of the sample mutually agreed upon for color by the buyer and seller in clear glass test tubes of the same diameter.

The color of the sample under examination shall not be darker than the color of the sample mutually agreed upon for color by the buyer and seller.

NOTE.—The sample used for color comparison should be kept in a glass container in a dark place.

3. REAGENTS.—(a) *Acetic acid*.—Ninety-nine per cent acetic acid having a melting point of 14.8° C., free from reducing impurities as shown by its action on bichromate-sulphuric acid mixture.

(b) *Wijs solution*.—Dissolve 13 g of pure iodine (resublimed grade) in a liter of acetic acid (see (a) above), using gentle heat if necessary. Cool and determine the strength by titration with thiosulphate solution (see (d) below). Set aside 50 to 100 cc of the solution and introduce dry chlorine gas into the remainder until a characteristic color change occurs and the halogen content has been doubled. By titration ascertain if the halogen content has been more than doubled, and, if so, reduce it by adding a requisite quantity of the iodine-acetic acid solution. The final product should contain an amount of iodine and chlorine corresponding to iodine-monochloride; a slight excess of iodine does no harm, but any excess of chlorine over the theory for ICl must be avoided.

(c) *Chloroform*.—U. S. P. chloroform should be used.

(d) *Sodium thiosulphate solution*.—Dissolve pure sodium thiosulphate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g crystallized sodium thiosulphate will be present in 1,000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Analytical Chemistry, Treadwell-Hall, Vol. II, 3d ed., p. 646.) This solution will be approximately decinormal, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly decinormal strength. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(e) *Starch solution*.—Dissolve 0.2 g of starch per 100 cc of distilled water and boil.

(f) *Potassium iodide solution*.—Dissolve potassium iodide free from iodate in distilled water in the proportion of 1 part salt to 10 parts water.

(g) *Ninety-five per cent alcohol*.—Either 95 per cent ethyl alcohol or 95 per cent specially denatured alcohol, United States Internal Revenue Bureau Formula No. 1 or No. 30. (Formula No. 1 calls for the addition to 100 gallons ethyl alcohol (190 proof) of 5 gallons approved wood alcohol, the wood alcohol to be subject to the same specifications as are imposed upon wood alcohol used in completely denatured alcohol. Formula No. 30 calls for the addition to 100 gallons ethyl alcohol of 190 proof of 10 gallons of pure methyl alcohol, which methyl alcohol is to have a specific gravity of not more than 0.810 at 60° F.)

VII. PACKING AND MARKING

Shellac varnish shall be shipped in containers that will not darken the product during storage.

VIII. NOTES

This specification is intended to cover all the needs of the Government for shellac varnish, both orange and bleached. Bleached shellac varnish is sometimes known as white shellac varnish. In general, type 1 of orange shellac varnish will include the rosin-free material and the lighter colored varnishes, while type 2 is intended for use where the presence of small percentages of rosin (not over 3 per cent) is not objectionable and where lightness of color is not an essential factor.

In general, type 1 of bleached shellac varnish will fulfill all the needs for a white shellac varnish. Type 2 will only be used in cases

where a clear bleached varnish (practically free from wax or other suspended matter) is necessary.

Light, medium, and heavy body varnishes correspond to 3.5, 4.0, and 4.5 pound "cuts" of shellac, respectively. The minimum allowable percentages of nonvolatile matter for each body of varnish given in Section V of this specification have been corrected for maximum moisture and volatile matter in flake orange shellac of 2 per cent and in bleached dry shellac of 5 per cent, so that varnishes made by cutting 3.5, 4.0, and 4.5 pounds of either orange or bleached shellac in 1 gallon of specially denatured alcohol should meet the nonvolatile requirements of Section V.

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